

CLEAN VERSION OF AMENDED SPECIFICATION

On page 25, please amend the paragraph beginning at line 24 as follows:

As shown in this table, when acetic anhydride is used in ring-opening-polymerizing THF has a diketene concentration of 10 ppm or less, hue decreases significantly as compared with PTMG produced by using acetic anhydride having a diketene concentration higher than this concentration, further, when acetic anhydride containing little diketene having a diketene concentration of the detection limit or less is used, colorless and transparent PTMG of high quality is obtained.



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CERTIFICATE OF TRANSLATION

I, YOSHIKAWA Toshio, with postal address at Murahama Bldg. 6F, 4-9-19, Miyakojima-ku, Higashinoda-cho, Osaka 534-0024 Japan, declare that I am well acquainted with the Japanese and English languages and that the attached specification, claims and abstract in English are true translation of those filed originally in Japanese on February 17, 2000, under the application number of 2000-039180.

Dated this 19th day of November, 2002



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RECEIVED
DEC 19 2002
TC 1700

【Document's Name】 Application for Patent

【Reference Number】 P000217-1

【Address】 To the Commissioner of the J.P.O.

【International Patent Classification】 C07C 53/12

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 【Payment】 Payment before filing application

 【Number of Register of Payment】 021360

 【Amount】 21000

【List of Attached Documents】

 【Document's Name】 Specification 1

【Document's Name】	Drawing	1
【Document's Name】	Abstract	1
【Power of Attorney Number】	9908796	

【Document's name】 Specification

【Title of the invention】 Method of purifying crude acetic anhydride, and method of producing polyoxytetramethylene glycol using acetic anhydride

【Claim】

【Claim 1】 A method of purifying crude acetic anhydride wherein treatment with an ozone-containing gas is conducted after distillation.

【Claim 2】 A method of producing polyoxytetramethylene glycol, which is characterized by producing polyoxytetramethylene glycol, by ring-opening-polymerizing tetrahydrofuran in the presence of the acetic anhydride purified by the purification method of Claim 1, and an acid catalyst.

【Detailed Description of the Invention】

【0 0 0 1】

【Field of the Invention】

The present invention relates to a method of purifying crude acetic anhydride, and a method of producing polyoxytetramethylene glycol using acetic anhydride.

【0 0 0 2】

【Prior Art】

As the industrial method of producing acetic anhydride, a method (Ketene process) in which acetic acid is thermally decomposed to produce a ketene, and acetic acid is allowed to absorb and react with this ketene, to obtain acetic anhydride, and a method in which carbon monoxide is allowed to react with methyl acetate or dimethyl ether to obtain acetic anhydride, as well as other methods are known.

【0 0 0 3】

When acetic anhydride is produced by a Ketene process, for example, acetic acid which was condensed to be collected from an aqueous acetic acid solution which was discharged from the production process of cellulose acetate is often used as its raw material of acetic acid, and an impurity derived from a cellulose system is also contained in the condensed acetic acid. Further, since various low boiling point compounds and high boiling point compounds which were

produced at the thermal decomposition reaction of acetic acid and the absorption reaction of ketene and acetic acid are contained, a purification processing which removes the low boiling point compounds and high boiling point compounds by distillation is carried out.

【0004】

With respect to the purification of crude acetic anhydride, since the consumption of thermal energy is great by distillation processing, for example, there is disclosed a purification method of treating crude acetic anhydride with an ozone-containing gas in place of distillation and removing impurity in Japanese Patent Publication No.4-34537 (hereinafter, referred to as JP4-34537B).

【0005】

On the other hand, a purification method of successively carrying out an ozone processing and distillation is disclosed in JP6-25071A, on the assumption that acetic acid which is obtained by contact carbonylation of methanol and/or methyl acetate and methanol and/or dimethyl ether, and acetic anhydride. Namely, when acetic acid and acetic anhydride are simultaneously produced by reaction of methyl acetate and the like and carbon monoxide, many unsaturated compounds are contained, and since the boiling point of methyl crotonate is about the same boiling point of acetic acid, it is difficult to remove them by a usual distillation. Further, when the unsaturated compounds are treated with the ozone processing, acetaldehyde is prepared which is a substance exerting a bad influence to the retention time of potassium permanganate test which is an index showing the quality of acetic acid and acetic anhydride. Accordingly, in the purification method described in the above-mentioned publication, there is adopted a purification method of contacting acetic acid and acetic anhydride with ozone and then distilling them.

【0006】

【Problems to be solved by the Invention】

The crude acetic anhydride which was obtained by the said method is purified by various purification methods, and, for example, when polyoxytetramethylene glycol (hereinafter, referred to as PTMG) which is industrially useful as a main raw material and the like of a

polyurethane, a poly(ether ester) and a poly(ether(ester) amide) is produced, there is generated a problem that a PTMG product having good hue is difficult to be stably obtained.

【0007】

PTMG is usually produced by carrying out the ring open polymerization of tetrahydrofuran in the presence of acetic anhydride and an acid catalyst to produce polyoxytetramethylene glycol diester, and then hydrolyzing the polyoxytetramethylene glycol diester in the presence of an alkali catalyst or carrying out ester exchange with a lower alcohol, for example, methanol. In the production of PTMG, even if either of those which was obtained by distilling acetic anhydride by the above-mentioned method, or those which was obtained by applying the processing method of the said JP6-25071A and distilling after the ozone processing are used, the obtained PTMG product is colored to be yellow, and there is generated a problem that the product having an adequate transparency is not stably obtained.

【0008】

The present invention was achieved considering the above-mentioned problem, and the purpose is to provide the purification method of crude acetic anhydride which can improve the quality at using it for production of PTMG and the like, and the production method of PTMG using acetic anhydride.

【0009】

【Means to solve the problems】

In order to attain the above-mentioned purpose, the present inventors have intensively studied the purification method of crude acetic anhydride and the quality of a product, for example, PTMG, which is produced using acetic anhydride, and as a result, have found that diketene which are contained in acetic anhydride greatly influences the quality of a product, to complete the present invention.

【0010】

Namely, the purification method of crude acetic anhydride of the present invention is characterized in that it is treated with an ozone-containing gas after crude acetic anhydride is distilled.

【0011】

Further, as described above, the production method of PTMG of the present invention is characterized in that polyoxytetramethylene glycol is produced by carrying out the ring open polymerization of tetrahydrofuran in the presence of an acid catalyst and acetic anhydride which is obtained by being treated with an ozone-containing gas after distilling crude acetic anhydride.

【0012】

【Embodiment of the invention】

The raw material crude acetic anhydride, which is used in the purification method of acetic anhydride of the present invention, may be crude acetic anhydride having any concentration in a process of producing acetic anhydride. However, when the amount of impurities in crude acetic anhydride obtained by the reaction process is higher, the amount of consumption of an ozone-containing gas described later increases, consequently, it is desirable to produce crude acetic anhydride by selecting reaction conditions under which the amount of double bond components such as for example diketenes and the like contained in the crude acetic anhydride is lower.

【0013】

First, the mode of a distillation column used in primary purification by distilling crude acetic anhydride is not particularly restricted, and can be selected freely. In general, it is possible to use one or more columns selected from plate columns such as a sieve tray, bubble cap tray, valve tray and the like, packed columns such as an interlocks saddle, ball ring, throughzer bag and the like.

【0014】

In the case of the plate column, tray number of about 20 to 80 is preferable, and in the case of the packed column, that having the corresponding packed height is preferably used. Acetic anhydride to be purified is introduced through the intermediate part of the distillation column, and it is necessary that the introduction position is upper than the position for recovering the produce acetic anhydride, and desirably, parts upper than the center position of the distillation column are selected. The product acetic anhydride is recovered in the form

of vapor or liquid from parts lower than the raw material introduction stage, desirably parts lower than the center position of the distillation column.

[0 0 1 5]

The operation pressure of the distillation column is not particularly restricted, and when the pressure is too high, there is a fear of occurrence of an undesirable reaction by increase in intra-column temperature, while, when the pressure is too low, condensation of vapor at the column top is followed by difficulty. Therefore, the desirable operation pressure is in the range from 100 mmHg to normal pressure at the column top.

[0 0 1 6]

A part of condensation liquid of the column top vapor is returned to the column top as reflux liquid, and the ratio of reflux liquid flow to the recovering liquid flow, namely reflux ratio is determined depending on the composition of the raw material liquid, required quality of the product, and the like. It can be selected usually in the range from about 1 to 1000.

[0 0 1 7]

Next, when crude acetic anhydride primary-purified using the above-described distillation column is secondary-purified by ozone treatment, the ozone-containing gas used in this procedure is also not restricted particularly. Industrially, a method is generally used in which ozone is generated by silent discharge using air or oxygen as a raw material. Usually, in the case of an air material, the ozone concentration is from 10 to 20 g/Nm³. Regarding the ratio of ozone to be contacted with acetic anhydride, it may be recommendable to charge somewhat excess amount of ozone for completely stop the reaction in view of a decomposition reaction of ozone itself, and the like. Practically, it is necessary to determine the ratio appropriately by experiments in view of the gas-liquid contact efficiency and purification efficiency, and usually, the ozone use ratio is from 50 to 300 g-O₃/T, preferably from 90 to 270 g-O₃/T.

[0 0 1 8]

The form of a reaction vessel for the ozone treatment is also not particularly restricted providing contact of ozone with acetic anhydride can be conducted successfully,

and practically, a bubble column mode and an agitating vessel mode are preferable. Further, as the contact time, suitable time in the range from decades seconds to dozens minutes may advantageously be set. The reaction temperature is suitably near room temperature, and preferably from about 20 to 30°C. When the temperature is too low, the reaction speed decreases, while when too high, ozone itself is not decomposed easily, undesirably.

【 0 0 1 9 】

In accordance with the embodiment above, purified acetic anhydride in which the content of diketene is detection limit (about 2ppm) or less can be obtained by carrying out the secondary purification by the ozone-containing gas after the primary purification by which crude acetic anhydride is distilled.

【 0 0 2 0 】

On the other hand, according to the production method of PTMG of the present invention, polyoxytetramethylene glycol diester (hereinafter, described as PTMGAC in abbreviation) is firstly produced by carrying out the ring open polymerization of tetrahydrofuran (hereinafter, described as THF in abbreviation) in the presence of an acid catalyst and purified acetic anhydride which is obtained as described above, and PTMG is produced from PTMGAC.

【 0 0 2 1 】

Acid catalyst used in this case is also not restricted particularly, and those public-known can be used. For example, solid acid catalysts such as super strong acidic cation exchange resin, bleached soil, zeolite and the like can be used. Liquid acids such as perchloric acid can also be used, however, this case is industrially disadvantageous since a process for neutralizing and/or separating an acid after the ring-opening-polymerization is necessary and becomes complicated. Use of a solid acid catalyst is preferable, since separation of a catalyst can be conducted simply in this case. A solid acid catalyst can be used in any of a suspension bed and fixed bed, and use of the catalyst in a fixed bed flow reaction is particularly preferable since it is not necessary to separately conduct a catalyst separation operation in this case.

【 0 0 2 2 】

The reaction conditions vary depending on the molecular weight of the intended PTMG and the kind of an acid catalyst used, and usually, the concentration of an acid catalyst in the reaction liquid is from 0.1 to 50% by weight, the concentration of acetic anhydride is set from about 0.1 to 30% by weight. The reaction time is usually selected in the range from 0.5 to 10 hours. Since the resulted polymerization reaction liquid contains PTMGAC and unreacted raw materials and the like, usually unreacted THF and acetic anhydride are distilled off under normal pressure or reduced pressure.

【0023】

Production of PTMG from this PTMGAC obtained as above is usually conducted by replacing an end ester group with a hydroxyl group by hydrolysis or alcoholysis in the presence of an alkali catalyst.

【0024】

First, production of PTMG by hydrolysis in the presence of an alkali catalyst will be described. This alkali hydrolysis is a method in which an alkali aqueous solution is added in the presence of an organic solvent, and heated to substitute an end ester group with a hydroxyl group. As the organic solvent, compounds which are separated from water, such as aromatic hydrocarbons like benzene, toluene, xylene and the like, aliphatic alcohols like n-butanol and the like, aliphatic ethers like diisopropyl ether and the like are used.

【0025】

As the alkali catalyst, hydroxides of alkali metals and alkaline earth metals such as sodium hydroxide, potassium hydroxide, calcium hydroxide and the like are used. These hydroxides are usually used as an aqueous solution. This aqueous solution is usually used in a concentration of 1 to 50% by weight.

【0026】

The amount of this aqueous solution added differs depending on the weight of water based on PTMGAC used, and usually, the aqueous solution is so used that the weight of water based on PTMGAC is 0.1 to 10-fold, and the concentration of an alkali based on the total weight of PTMGAC, water and the alkali is from 0.01 to 40% by weight. The alkali

hydrolysis temperature differs depending on the alkali concentration and the kind of an alkali used, and usually in the range from 50 to 150°C. Further, also alkali hydrolysis time differs depending on the alkali concentration, the kind of an alkali used, and the hydrolysis temperature, and usually, in the range from 0.1 to 20 hours.

【0027】

The reaction crude solution containing water after completion of the alkali hydrolysis is separated into an organic layer and aqueous layer for example by a centrifugal separator, and an organic solvent and water are evaporated from the recovered organic layer by using an evaporator having a short residence time such as a thin layer evaporator, to obtain the product PTMG.

【0028】

Then, production of PTMG from PTMGAC by substituting an end ester group with a hydroxyl group by alcoholysis in the presence of an alkali catalyst will be described.

【0029】

The alcoholysis is a method in which PTMG is produced while extracting the by-produced carboxylate by azeotrope with alcohol by reaction distillation, using aliphatic alcohol such as methanol and the like. As this alcohol, ethanol, butanol and the like are used in addition to methanol. Among them, methanol is most preferable from the standpoints of cost, reactivity, and separation property between raw material alcohol and an ester produced by the reaction.

【0030】

As the alkali catalyst, alkaline earth metal oxides, or alcoholates of alkali metals or alkaline earth metal are used. As the alkaline earth metal oxide, magnesium oxide, calcium oxide and strontium oxide are preferable. This catalyst is usually used in the form of a powder, and may also be used in the tablet form, and the form may appropriately be selected advantageously by selection of the reaction mode and catalyst separation method.

【0031】

The amount of the catalyst used is usually from 0.1 to 10% by weight, more

preferably from 0.5 to 3% by weight in terms of the concentration of the catalyst in the reaction crude solution. When the catalyst is a powder, the catalyst concentration may also be lower since the contact surface area per unit weight of the catalyst with reaction crude solution is large, and in the case of a catalyst which has been tabletted and molded, higher catalyst concentration is preferable wince the contact area per unit weight of the catalyst is small.

【0032】

On the other hand, when an alcoholate of alkali metals or alkaline earth metals is used as the catalyst in alcoholysis, the catalyst concentration differs depending on the kind of the catalyst, and from 0.01 to 3% by weight. Further, the catalyst is used in the form of an alcohol solution from the standpoint of handling easiness.

【0033】

The reaction temperature of alcoholysis is not particularly restricted, and usually from 30 to 120°C under normal pressure. In the case of a reaction with alcohol in which the reaction temperature is over 120°C under normal pressure, the final product gets poor hue, therefore, it is preferable, in such a case, to conduct the reaction under reduced pressure so that the reaction temperature in a vessel of a distillation column is 120°C. On the other hand, when the reaction temperature is too low, the time before completion of the reaction is too long, undesirably. Further, when alcohol having lower boiling point such as methanol, ethanol and the like is used, the reaction may also be conducted under positive pressure to increase the reaction temperature in a vessel of a distillation column and to shorten the time before completion of the reaction.

【0034】

Alcoholysis may be conducted in batch-wise mode or continuous mode, and in the case of the batch-wise mode, a catalyst, PTMGAC and alcohol are charged in a vessel of a batch distillation column equipped with a reflux apparatus and reaction distillation is conducted, carboxylates produced in the vessel is distilled off by azeotrope with alcohol, then, a reaction is conducted until the column top temperature reaches the boiling point of the

alcohol, to complete the alcoholysis.

【 0 0 3 5 】

In the case of the continuous mode, a catalyst, PTMGAC and alcohol are continuously charged into a continuous distillation column so as to obtain residence time to complete the reaction, the produced carboxylate is continuously extracted in the form of an azeotropic mixture with the raw material alcohol from the top of the continuous distillation column, and the unreacted alcohol, produced PTMG and catalyst are extracted continuously from the vessel. The distillation column, in this case, differs depending on the kind thereof, fillers and distillation mode (continuous distillation, batch-wise distillation), and it is preferable to use a distillation column having a theoretical stage number of 20 to 100.

【 0 0 3 6 】

When a distillation column having lower theoretical stage number is used, separation between raw material alcohol and an azeotropic mixture of the raw material alcohol with the produced carboxylate is difficult. Usually, since an azeotropic mixture of raw material alcohol with the produced carboxylate is burnt, when separation between raw material alcohol and an azeotropic mixture of the raw material alcohol with the produced carboxylate is poor, the amount of the raw material alcohol used increases, indicating undesirable phenomenon. On the other hand, when a distillation column having higher theoretical stage number is used, since more stages than necessary for separation between raw material alcohol and an azeotropic mixture of the raw material alcohol with the produced carboxylate are present, equipment cost increases and also running cost increases.

【 0 0 3 7 】

The reaction time (residence time) of alcoholysis is determined by the catalyst temperature, reaction temperature and the molar ratio of raw material alcohol to PTMGAC, and usually in the range from 1 to 10 hours. When the reaction time is too long, excess residence time is left even if the reaction is terminated, leading to reduction in the production amount of PTMG, while when the reaction time is too short, the production reaction of PTMG by alcoholysis of PTMGAC is not completed. Since an ester residue in PTMG

deteriorates the quality of a product such as polyurethane and the like produced from PTMG, it is necessary that the reaction to produce PTMG from PTMGAC is completed.

【0038】

The molar ratio of PTMGAC to alcohol used in alcoholysis differs also depending on the number-average molecular weight and degree of dispersion of PTMGAC, and usually the molar ratio of alcohol to PTMGAC is selected in the range from 3 to 100. When the molar ratio of alcohol to PTMGAC is too low, alcoholysis is delayed significantly and the reaction time is elongated, additionally, there is a fear of no completion of the reaction, meaning undesirable phenomenon. On the other hand, when the molar ratio of alcohol to PTMGAC is too high, energy cost required for alcoholysis increases and the amount of alcohol flushed after alcoholysis increases, leading to increase in energy cost, undesirably.

【0039】

When there is a catalyst insoluble in the reaction crude solution from the bottom product of a distillation column produced by alcoholysis of PTMGAC in the presence of an alkaline earth metal oxide catalyst, the catalyst is usually separated and removed by filtration separation or centrifugal separation, and to recover and recycle unreacted alcohol, flushing is effected using a flushing apparatus having shorter residence time such as a thin layer evaporator and the like.

【0040】

Crude PTMG obtained from the bottom of a thin layer evaporator contains a catalyst component dissolved in the reaction crude solution, and to remove these catalyst component and oligomer, water and crude PTMG are charged in a stirring vessel, and stirred for several hours while heating. After completion of washing while stirring, the mixture is separated by a centrifugal separator, then, separated into an aqueous layer and an organic layer and the organic layer is recovered, and the organic solvent and water are evaporated by using an evaporator having shorter residence time such as a thin layer evaporator, to obtain the product PTMG.

【0041】

By using purified acetic anhydride diketene concentration of less than detection limit as the acetic anhydride used in a process to first produce PTMG by ring-opening-polymerizing THF, when PTMG is produced according to the embodiment as described above, PTMG having excellent hue can be obtained.

【0042】

【Examples】

Hereinafter, the following will illustrate an example of the experiments which investigate the content of diketene in purified acetic anhydride achieved by distilling crude acetic anhydride obtained by Ketene method, for example, then treating the distilled product with an ozone-containing gas, but it does not limit the scope of the present invention.

【0043】

First, a ketene obtained by thermal decomposition of acetic acid through a ketene furnace was absorbed in acetic acid to obtain crude acetic anhydride, this crude acetic anhydride was passed sequentially through a lower boiling point fraction-removing column and a higher boiling point fraction-removing column each operation pressure of which was maintained at normal pressure, to conduct primary-purification treatment. Then, ozone treatment was conducted on the crude acetic anhydride subjected to this primary purification treatment using an experimental apparatus shown in Fig. 1 as the second purification treatment, and the diketene concentration in the product acetic anhydride was measured under the varied ozonizing conditions.

【0044】

In Fig. 1, 1 represents an ozone generator, and 2 represents a packed column. Ozone was generated from the ozone generator 1 using air as a raw material, and ozonized air (mixed gas of ozone and air) discharged from this ozone generator 1 was introduced to the packed column 2 equipped with a raschig ring of internal diameter 5 mm × height 5 mm through the lower part thereof. On the other hand, the crude acetic anhydride subjected to the primary-purification treatment by distillation as described above was charged into the packed column 2 through the upper part, and allowed to contact countercurrent with ozone, to

effect ozone treatment. The product acetic anhydride subjected to the ozone treatment was extracted through the lower part of the packed column 2, and recovered. The temperature of packed column is 30 degree centigrade and the pressure is normal.

[0 0 4 5]

The measuring results of the concentration of diketene in changing the ozone inflow amount to the packed column 2 in the product acetic anhydride are shown in Table 1.

[0 0 4 6]

As shown in this table, in the Experiment 1, the charge flow amount of the crude acetic anhydride subjected to the primary-purification treatment was 6279 g/H, the ozone inflow amount was 27.4 mol/H, and the ozone use ratio in this case was 93.6 g-O₃/T. In the Experiment 2, the charge flow amount of the crude acetic anhydride was, almost same as the above, 6006 g/H, the ozone inflow amount was 38.3 mol/H, and the ozone use ratio in this case was 134.3 g-O₃/T. Furthermore, the concentration of diketene in the crude acetic anhydride is 76 ppm in either case. As shown in these results, when the ozone used ratio is over 90 g-O₃/T, the diketene concentration was the detection limit (2 ppm) or less.

【0047】

[Table 1]

Experiment No.	#1	#2
Amount of ozonized air (NL/H)	30	42
Concentration of ozone (g/Nm ³)	19.0	←
Flow-in amount of ozone (mol/H)	27.4	38.3
Charge amount of acetic anhydride (g/H)	6279	6006
Concentration of diketene (ppm)	76	←
Usage rate of ozone (g-O ₃ /T)	93.6	134.3
Concentration of diketene in acetic anhydride of finished product (ppm)	ND	ND

ND: Detection limit (2ppm) or less

【0048】

On the contrary to the above, the diketene concentration in the product acetic anhydride when purification treatment was conducted according to a purification method (hereinafter, referred to as the conventional method) in which ozone treatment is conducted previously, then, distillation treatment is conducted through the lower boiling point fraction-removing column and the higher boiling point fraction-removing column was at least about 5 ppm. Therefore, according to the present invention, when a purification method in which distillation treatment is conducted previously, then, ozone treatment is conducted is used, the content of diketenes is the detection limit or less, and a product acetic anhydride having more high quality which was impossible by the conventional method can be produced.

【0049】

On the other hand, when PTMG is produced by carrying out the ring open polymerization of tetrahydrofuran in the presence of acetic anhydride and a solid acid catalyst and carrying out the ester exchange of PTMGAC which was obtained by this, with methanol in the presence of an alkali catalyst, PTMG which was produced using acetic anhydride which was obtained by a conventional method described above, as acetic anhydride, was colored in slight

yellow, but PTMG which was produced using the purified acetic anhydride which was obtained in accordance with the method of the present invention in which diketene is the under detection limit was nearly clear and colorless, therefore a PTMG product having a good hue was obtained.

【0050】

【Effect of the invention】

As discussed above, according to the present invention, crude acetic anhydride having smaller diketene content can be obtained. By using acetic anhydride having such small diketene concentration, products such as PTMG and the like having excellent hue can be produced.

【Brief description of the drawing】

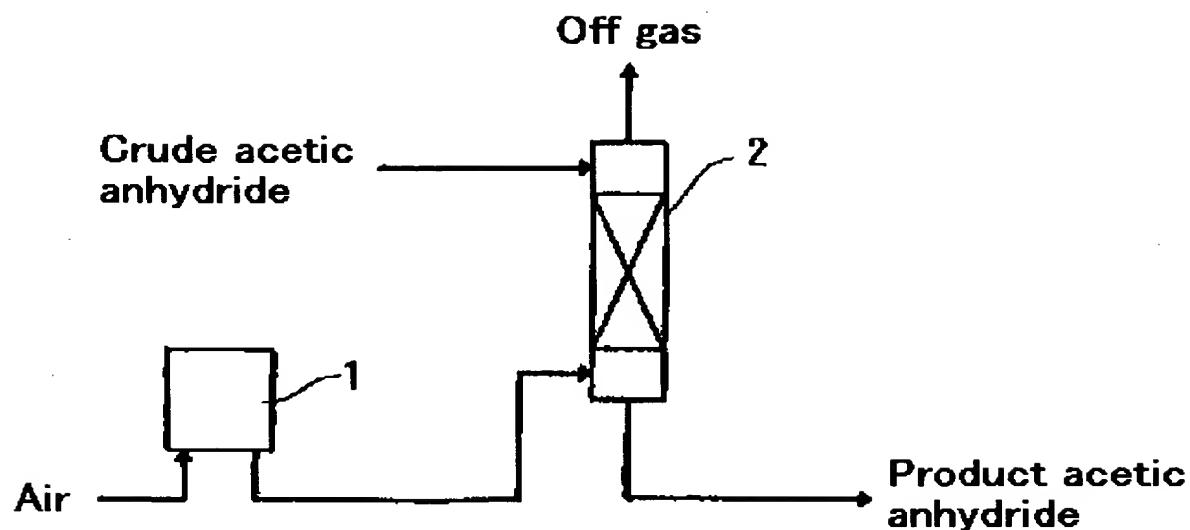
Fig. 1 is a schematic view showing the constitution of an apparatus used in conducting ozone treatment on crude acetic anhydride according to the present invention.

【Description of the code】

1. an ozone generator
2. a packed column

[Document's Name] Drawing

[Fig. 1]



【Document's Name】 Abstract

【Abstract】

【Problem to be solved】 To provide a method for purifying the crude acetic anhydride, giving a purified acetic anhydride which can improve quality of polyoxytetramethylene glycol (PTMG), etc.

【Solution】 This method for purifying the crude acetic anhydride comprises distilling the crude acetic anhydride, then charging a packed tower 2 with the distilled crude acetic anhydride together with ozone-containing air generated by an ozone generator 1, and further subjecting the charged mixture to ozonization. Thus the purified acetic anhydride of which the diketene content is not higher than the detection limit can be obtained, because the acetic anhydride is subjected to ozonization after the distillation. The PTMG which is almost colorless, transparent, and excellent in color can be produced by using the purified acetic anhydride.

【Selected Drawing】 Fig. 1